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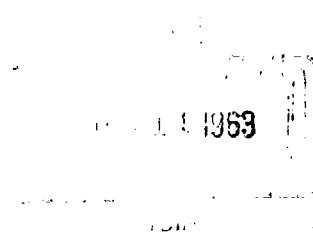
COATING and CHEMICAL LABORATORY

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FURTHER STUDIES OF DETERGENCY

Report No. CCL # 133
OMS Code No. 5010.11.8420051
D. A. Project No. 593-32-007
Author A. Mankowich
Date 19 November 1962



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By

A. Mankowich

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Dept of the Army Project No.
593-32-007

Coating and Chemical Laboratory
Aberdeen Proving Ground
Maryland

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ABSTRACT

Further study of the proposed (1) detergency (D) - micellar solubilization (S) function, $D = K_1 S + K_2$, centered around the connection of the constants K_1 and K_2 to physicochemical factors of soil and surfactant, has resulted in establishing K_1 and K_2 relationships for 4-point (4 surfactants-1 soil, and 4 soils - 1 surfactant) and 5-point (5 surfactants - 1 soil) systems. The constants appear to be related to physical, and not random, parameters.

Trends in the derived K_1 and K_2 relationships are (a) K_2 values of both multi-surfactant and multi-soil systems are functions of Antonow's interfacial tension, (b) K_1 values of multi-surfactant systems are connected to surfactant HLB, while K_1 values of multi-soil systems are connected to soil dipole moment, (c) prevalence of trigonometric functions especially in 5-point systems.

I. INTRODUCTION

Studies of detergency at this Laboratory (1, 2, 3, 4) have established that (a) a linear relationship ($D = K_1 S + K_2$) exists between dynamic hard surface detergency (D) and dynamic micellar solubilization of Orange OT dyestuff (S), between the concentration at about 90% soil removal (CC-1 point) and that at twice the latter point (CC-2 concentration), (b) the relationship has thus far been found to apply to four types of soil (fatty acid, alcohol, ester and amine), (c) the constant K_1 is a function of surfactant HLB (hydrophile-lipophile balance) and soil dipole moment, and K_2 is a function of the boundary tensions, for systems of four homologous surfactants and one soil or for systems of four classes of soil and one surfactant, (d) mathematically the K_1 and K_2 functions are probably physical rather than random because they contain fewer constants than the number of points (four) used in their derivation (4).

This report presents derivations of K_1 and K_2 relationships (a) for two 5-point systems (soil-surfactant), and (b) for additional 4-point systems; it also examines the K_1 and K_2 relationships established thus far for patterns in type of function and variables.

II. DETAILS OF TEST

A. Experimental

The details of the dynamic methods used for determining detergency and micellar solubilization, both at 180°F, have been given in earlier papers (2,5).

B. Materials

The surfactants were 100% active, commercial products, the same as those used in previous work (5,6). Included were the 20, 30, 40, 50 and 100 ethylene oxide mole ratio (R) adducts of nonyl phenol; namely,

20 R adduct	-----	NPEGE
30 R adduct	-----	NPTGE
40 R adduct	-----	NPTTGE
50 R adduct	-----	NP50E
100 R adduct	--	NP100E

The nonionic, polyoxyethylene-23 sorbitan monolaurate, PSML, was also studied.

Four of the soils (triolein, olein acid, lauryl alcohol, and octadecylamine) have been described previously (1, 2, 5). A purified grade of linoleic acid was also used as a soil.

C. Detergency Function

Although over long concentration ranges (3 to 4 multiple spreads) at 100°F, the log solubilization-log concentration function is linear (7), similar data at 180°F (5) revealed the linearity of the simple solubilization-concentration function in the shorter CC-1 to CC-2 range. In the latter range, the detergency-concentration relationship is linear within the precision of the

gravimetric detergency method. Since both detergency and micellar solubilization are linear with respect to concentration in the CC-1 to CC-2 range, each with a positive slope, it follows that the detergency-micellar solubilization function is also linear (1).

III. RESULTS AND DISCUSSION

A. Oleic Acid Soil With the Ethylene Oxide Adducts of Nonyl Phenol

Former reports (2, 6) give the micellar solubilization and oleic acid detergency data, together with the derived detergency-solubilization equations, for the 20, 30, 50, and 100 R adducts of nonyl phenol; viz;

Adduct	R	Equation
NPEGE	20	$D = 1.31 S + 85.75$
NPTGE	30	$D = 3.52 S + 91.52$
NP50E	50	$D = 1.66 S + 82.62$
NP100E	100	$D = 0.60 S + 89.06$

Table I lists the solubilization and oleic acid detergency data for the 40 R adduct, NPTTGE, from which the following equation may be obtained as previously described (6):

$$D = 7.65 S + 90.14$$

Since the HLB values of the 20, 30, 40, 50, and 100 R adducts are 16.00, 17.20, 17.78, 18.18, and 19.05, respectively (1/5 of their oxyethylene weight percentages), it can be shown that K_1 is a trigonometric function of HLB, such that:

$$K_1 = 1.95 - 5.95 \sin (3.12 \text{ HLB})$$

Agreement between calculated and experimental values of K_1 and of D_1 (detergency at the CC-1 point) is good for the 30, 40, 50, and 100 R adducts, as follows:

R	HLB	Calculated K_1	Experimental K_1	Calculated D_1^*	Experimental D_1
30	17.20	3.46	3.52	92.9%	91.9%
40	17.78	7.19	7.65	91.5%	91.6%
50	18.18	0.93	1.66	87.3%	89.7%
100	19.05	0.45	0.60	92.4%	90.1%

* Based on use of calculated K_1 in $D - S$ equations.

The K_2 boundary tension function for this system can be determined from the following applicable information:

R	K_2	Tensions, at CC-1 points, dynes/cm	
		Surface tension	Antonow's interfacial tension, A
20	85.75	35.8	4.3
30	91.52	37.7	6.2
40	90.14	39.0	7.5
50	82.62	44.6	13.1
100	89.06	46.3	14.8

Antonow's interfacial tension values, above, are differences between surfactant and soil surface tensions, not mutually saturated, at ca 28°C, with the surface tension of oleic acid taken as 31.5 dynes per cm. Strictly, the values are not interfacial tensions as defined by Antonow's rule, but they are specific factors. It follows that K_2 is a trigonometric function of A:

$$K_2 = 88.1 + 5.5 \sin (1.28 A)$$

The agreement between calculated and experimental values of K_2 is good for all five adducts, as follows:

R	A	Calculated K_2	Experimental K_2
20	4.3	84.2	85.75
30	6.2	93.6	91.52
40	7.5	87.1	90.14
50	13.1	83.2	82.62
100	14.8	88.7	89.06

The establishment of a function connecting the K_2 values (for oleic acid soil systems) of the five polyoxyethylated nonyl phenols with R values varying from 20 - 100 is considered important, particularly since the variables are simple physicochemical properties of the surfactants and the soil. Similarly, the relationship of the K_1 values of the detergency-solubilization function for the same homologous surfactants and soil, with R varying from 30 - 100 (four adducts), to HLB of the surfactants is significant.

B. Lauryl Alcohol Soil With Five Ethylene Oxide Adducts of Nonyl Phenol

A previous paper (4) gives the detergency and solubilization data for the lauryl alcohol soil - 20 R adduct system, and computes the following detergency function therefor:

$$D = 5.20 S + 87.83$$

Table I lists the lauryl alcohol detergency and applicable micellar solubilization data for the 30, 40, 50, and 100 R adducts of nonyl phenol, from which the following detergency functions may be calculated as previously described (6):

30 R Adduct	-----	$D = 20.00 S + 89.70$
40 R Adduct	-----	$D = 11.71 S + 90.71$
50 R Adduct	-----	$D = 128.57 S + 85.09$
100 R Adduct	-----	$D = 15.28 S + 89.47$

Using the HLB values given in section III-A, it follows that

$$K_1 = 67 + 62 \sin (10.485 \text{ HLB})$$

Agreement between calculated and experimental values of K_1 and of D_1 is fair to good for all five adducts; viz;

R	HLB	Calculated K_1	Experimental K_1	Calculated D_1^*	Experimental D_1
20	16.00	8.1	5.20	94.3%	90.2%
30	17.20	7.8	20.00	90.5%	91.8%
40	17.78	12.7	11.71	92.1%	91.9%
50	18.18	119.9	128.57	92.3%	92.8%
100	19.05	6.9	15.28	90.8%	92.3%

* Using calculated K_1 in D - S equations.

The relationship of K_2 to boundary tensions can be determined from an analysis of the following applicable information:

R	K_2	Tensions, at CC-1 points, dynes/cm	
		Surface tension	Antonow's interfacial tension, A^*
20	87.83	34.8	6.5
30	89.70	36.7	8.4
40	90.71	37.1	8.8
50	85.09	39.7	11.4
100	89.47	42.6	14.3

* Based on surface tension of lauryl alcohol at ca 28°C = 28.3 dynes/cm.

It is found that the K_2 - A function is trigonometric, as follows:

$$K_2 = 88.65 + 3.65 \sin (4.40 A)$$

Agreement between calculated and experimental values of K_2 is good for all five adducts; viz.,

R	A	Calculated K_2	Experimental K_2
20	6.5	87.5	87.83
30	8.4	86.2	89.70
40	8.8	91.8	90.71
50	11.4	88.3	85.09
100	14.3	89.0	89.47

C. NPEGE Detergency of Four Classes of Soil

NPEGE solubilization and triolein detergency data have been reported (5) and the corresponding detergency function calculated (6); viz.,

$$D = 2.55 S + 80.09$$

The NPEGE oleic acid detergency function has also been determined (section III-A):

$$D = 1.31 S + 85.75$$

Similarly, the NPEGE lauryl alcohol detergency function has been derived (section III-B):

$$D = 5.20 S + 87.83$$

Table I gives the octadecylamine detergency and applicable micellar solubilization data for NPEGE, the 20 R adduct of nonyl phenol, from which the following detergency function can be computed:

$$D = 0.36 S + 89.58$$

Analysis of the K_1 values and the corresponding soil dipole moments (given below) indicates approximate conformance to the following equation:

$$K_1 = 3.00 - 3.05 \sin (6.283 \text{ DM})$$

where, DM = dipole moment in debyes

Agreement between experimental values of D_1 and values computed from the detergency functions using the calculated K_1 figures is fair to good for most of the soils, as shown below:

Soil	DM*	Calculated K_1	Experimental K_1	Calculated D_1	Experimental D_1
Triolein	3.08	1.5	2.55	85.8%	89.6%
Oleic Acid	1.009	3.0	1.31	98.4%	90.0%
Lauryl Alcohol	1.7	5.9	5.20	92.6%	90.2%
Octadecylamine	1.3	0.10	0.36	91.0%	92.6%

* Dipole moments of triolein, oleic acid and lauryl alcohol are referenced in (6); dipole moment of octadecylamine is referenced in (4).

From the K_2 - boundary tension information below, a trigonometric K_2 - A function can be derived:

Soil	K_2	Soil Surface Tension	Tensions, at CC-1 points, dynes/cm	
			Surface tension	Antonow's tension, A
Triolein	80.09	31.6	35.9	4.3
Oleic Acid	85.75	31.5	36.4	4.9
Lauryl Alcohol	87.83	28.3	34.8	6.5
Octadecylamine	89.58	21.7	35.8	14.1

The surface tension of octadecylamine has been estimated from its critical surface tension of wetting, as previously described (2). Mathematical analysis indicates that:

$$K_2 = 87.30 + 7.20 \sin (2.43 A)$$

Agreement between calculated and experimental values of K_2 is good for all four soils; viz.,

Soil	A	Calculated K_2	Experimental K_2
Triolein	4.3	81.2	80.09
Oleic Acid	4.9	82.9	85.75
Lauryl Alcohol	6.5	86.7	87.83
Octadecylamine	14.1	89.4	89.58

D. PSML Detergency of Four Classes of Soil

PSML, polyoxyethylene-23 sorbitan monolaurate, solubilization and triolein soil removal data have been reported (5), and the corresponding detergency function determined (6); viz.,

$$D = 0.91 S + 86.44$$

Oleic acid detergency by PSML is given in Table I and pertinent solubilization data has been reported (5). The following detergency function may be calculated:

$$D = 1.33 S + 89.01$$

Table I contains detergency and micellar solubilization data for the PSML-lauryl alcohol and PSML-octadecylamine systems. The following detergency functions may be calculated:

$$\begin{aligned} \text{lauryl alcohol soil} & \text{----- } D = 8.14 S + 82.60 \\ \text{octadecylamine soil} & \text{----- } D = 0.13 S + 92.10 \end{aligned}$$

Mathematical analysis of the dipole moments of the aforementioned soils (given in section III-C) together with the K_1 values of their detergency functions reveals a trigonometric relationship, as follows:

$$K_1 = 2.80 - 3.00 \sin (6.283 \text{ DM})$$

Again, agreement between experimental values of D_1 and values computed from the respective detergency functions using the calculated K_1 figures is fair to good for most soils:

Soil	DM	Calculated K_1	Experimental K_1	Calculated D_1	Experimental D_1
Triolein	3.08	1.35	0.91	95.6%	92.6%
Oleic Acid	1.009	2.8	1.33	98.3%	91.5%
Lauryl Alcohol	1.7	5.65	8.14	88.3%	88.8%
Octadecylamine	1.3	- 0.05	0.13	91.0%	92.9%

The similarity of the K_1 - DM functions of the PSML - soils and NPEGE -soils systems is to be noted. It is interesting that, although different chemically, the two surfactants have some physicochemical similarities; namely, HLB values PSML and NPEGE are 16.70 and 16.00, respectively, while interfacial tensions and adhesion tensions of the two surfactants against triolein are 0.3 and 31.3

dynes/cm, respectively, at their CC-1 concentrations.

The connection of K_2 to boundary tensions for the PSML-soils system may be determined from analysis of the following pertinent information:

Soil	K_2	Soil Surface Tension	Tensions at CC-1 points, dynes/cm	
			Surface Tension	Antonow's Tension, A
Triolein	86.44	31.6	34.1	2.5
Oleic Acid	89.01	31.5	34.1	2.6
Lauryl Alcohol	82.60	28.3	33.9	5.6
Octadecylamine	92.10	21.7	33.8	12.1

It is found that a parabolic relationship exists; namely,

$$K_2 = 98.73 - 5.24 A + 0.379 A^2$$

Agreement between calculated and experimental values of K_2 is good; viz.,

Soil	A	Calculated K_2	Experimental K_2
Triolein	2.5	88.0	86.44
Oleic Acid	2.6	87.7	89.01
Lauryl Alcohol	5.6	81.3	82.60
Octadecylamine	12.1	90.8	92.10

E. Octadecylamine Soil With Ethylene Oxide Adducts of Nonyl Phenol

Table I lists the detergency and applicable micellar solubilization data for the 20, 30, 40 and 50 R adducts of nonyl phenol with octadecylamine soil. From these data the following detergency functions may be determined:

$$\begin{aligned} 20 \text{ R adduct} & \text{-----} D = 0.36 S + 89.58 \\ 30 \text{ R adduct} & \text{-----} D = 4.16 S + 88.82 \\ 40 \text{ R adduct} & \text{-----} D = 3.87 S + 88.38 \\ 50 \text{ R adduct} & \text{-----} D = 4.25 S + 80.76 \end{aligned}$$

The 100 R adduct, NP100E, had no CC-1 point with octadecylamine soil, maximum detergency being 55% in 0.00800 M solution.

Using the corresponding HLB values of the adducts given in section III-A, it can be shown that the K_1 values of the preceding detergency functions are related to HLB by the following parabolic equation:

$$K_1 = -402.76 + 45.93 (\text{HLB}) - 1.296 (\text{HLB})^2$$

Agreement between calculated and experimental values of K_1 and of D_1 is good:

R	HLB	Calculated K_1	Experimental K_1	Calculated* D_1	Experimental D_1
20	16.00	0.34	0.36	94.4%	92.6%
30	17.20	3.83	4.16	93.0%	91.4%
40	17.78	4.18	3.87	92.7%	90.0%
50	18.18	3.91	4.25	88.4%	88.5%

*Based on calculated values of K_1 .

Mathematical analysis of the following K_2 -boundary tension factors also results in a parabolic relationship:

R	K_2	Tensions at CC-1 points, dynes/cm	
		Surface Tension	Antonow's Tension, A *
20	89.58	35.8	14.1
30	88.82	39.6	17.9
40	88.38	41.3	19.6
50	80.76	43.3	21.6

*Based on surface tension of octadecylamine = 21.7 dynes/cm

$$\text{Function: } K_2 = 25.74 + 8.25 A - 0.264 A^2$$

Agreement between calculated and experimental values of K_2 is excellent:

R	A	Calculated K_2	Experimental K_2
20	14.1	89.58	89.58
30	17.9	88.82	88.82
40	19.6	86.02	88.38
50	21.6	80.77	80.76

F. Linoleic Acid Soil With Ethylene Oxide Adducts of Nonyl Phenol

The following detergency functions may be computed from the linoleic acid detergency and solubilization data for the ethylene oxide adducts of nonyl phenol:

$$\begin{aligned} 30 \text{ R Adduct} & \text{----- } D = 5.90 S + 85.21 \\ 40 \text{ R Adduct} & \text{----- } D = 4.00 S + 90.36 \\ 50 \text{ R Adduct} & \text{----- } D = 2.95 S + 87.77 \\ 100 \text{ R Adduct} & \text{----- } D = 1.90 S + 89.26 \end{aligned}$$

Examination of the above K_1 values together with the corresponding HLB values reveals that the $\log K_1$ - HLB function is linear, as follows:

$$K_1 = \text{antilog } (5.2755 - 0.2625 \text{ HLB})$$

Agreement between calculated and experimental values of K_1 is good to excellent:

R	HLB	Calculated K_1	Experimental K_1
30	17-20	5.76	5.90
40	17.78	4.06	4.00
50	18.18	3.19	2.95
100	19.05	1.88	1.90

The connection of K_2 boundary tensions for this system may be determined from an analysis of the following pertinent information:

R	K_2	Tensions at CC-1 points, dynes/cm	
		Surface Tension	Antonow's Tension, A^*
30	85.21	39.3	7.6
40	90.36	39.1	7.4
50	87.77	43.5	11.8
100	89.26	45.0	13.3

*Based on surface tension of linoleic acid = 31.7 dynes/cm at 28.3°C.

It is found that the relationship is parabolic; viz.,

$$K_2 = 118.11 - 5.733 A + 0.268A^2$$

Agreement between calculated and experimental values of K_2 is good:

R	A	Calculated K_2	Experimental K_2
30	7.6	90.02	85.21
40	7.4	90.37	90.36
50	11.8	87.78	87.77
100	13.3	89.27	89.26

G. Discussion

Study of the detergency - micellar solubilization function, $D = K_1 S + K_2$, proposed by this laboratory (1) has centered around the relationship of the constants K_1 and K_2 to certain physicochemical factors of the soils and surfactants involved. Initial investigations (1) were of 3-point systems (3 surfactants with the same soil, or 3 soils with the same surfactant), for which no special effort was made to derive mathematically physical equations for K_1 and K_2 . Some such equations contained three constants, and since they applied to 3-point systems, the relationships could have been random. The significance of the concept has now been advanced not only by its application to 4-point and 5-point systems, but by the derivation of K_1 and K_2 equations containing fewer constants than the points of their system, which tends to make the relationships more probably physical. The latter is particularly true of the K_2 equations, for which agreement between calculated and experimental values is excellent.

In addition, the K_1 and K_2 relationships obtained so far are characterized by patterns the continued presence of which in subsequent systems studied will enhance the value of the developed detergency function. Trends noted include (a) K_2 values of both multi-soil and multi-surfactant systems are connected to Antonow's interfacial tension, the difference between surfactant and soil surface tensions, (b) K_1 values of multi-surfactant systems have been found to be functions of surfactant HLB, while K_1 values of multi-soil systems have been related to soil dipole moment. Also of interest at this point is the prevalence of trigonometric K_1 and K_2 functions, especially in the 5-point systems studied. Because of the limited number of systems investigated, these trends should of course be viewed with caution at this time.

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APPENDIX

Tables

TABLE I

DETERGENCY - MICELLAR SOLUBILIZATION DATA

Soil	Surfactant	Molarity	% Soil Removal	Micellar Solubilization, mg Orange OT per 100 ml
Oleic acid	NPTTGE	.000202	91.6	0.185
		.000404	96.6	0.845
Lauryl alcohol	NPTGE	.000138	91.8	0.105
		.000207	94.9	0.255
		.000275	97.6	0.395
Lauryl alcohol	NPTTGE	.000113	91.9	0.110
		.000225	93.3	0.215
Lauryl alcohol	NP50E	.000099	92.8	0.060
		.000149	95.0	0.080
		.000197	97.3	0.095
Lauryl alcohol	NP100E	.000250	92.3	0.185
		.000500	97.8	0.545
Octadecylamine	NPEGE	.00636	92.6	14.1
		.00954	99.0	21.5
		.0127	99.3	27.3
Oleic acid	PSML	.00131	91.5	1.9 at .000850 M
		.00170	96.8	4.5
		.00261	98.3	6.8 at .00255 M
Lauryl alcohol	PSML	.000424	88.8	1.0 at .000425 M
		.000571	95.5	1.35
		.000848	97.9	1.9 at .000850 M
Octadecylamine	PSML	.0106	92.9	22.5
		.0159	99.5	38.4
		.0212	99.6	56.3
Octadecylamine	NPTGE	.000455	91.4	1.10
		.000682	98.1	1.75
		.000909	99.0	2.45
Octadecylamine	NPTTGE	.000505	90.0	1.04
		.000758	97.1	1.65
		.00101	97.2	2.28
Octadecylamine	NP50E	.000788	88.5	1.96
		.00118	93.4	2.83
		.00158	96.8	3.77

TABLE I - CONTINUED

DETERGENCY - MICELLAR SOLUBILIZATION DATA

Soil	Surfactant	Molarity	% Soil Removal	Micellar Solubilization, mg Orange OT per 100 ml
Linoleic acid	NPTGE	.000413	89.2	0.98
		.000550	95.2	1.38
		.000826	98.2	2.20
Linoleic acid	NPTTGE	.000303	92.0	0.50
		.000450	93.9	0.95
		.000606	95.4	1.26
Linoleic acid	NP50E	.000661	91.8	1.50
		.000788	93.5	1.96
		.00132	97.1	3.16
Linoleic acid	NP100E	.00100	92.4	1.87
		.00150	95.3	2.95
		.00200	96.8	3.98

TABLE II

SURFACTANT NOMENCLATURE

Name	Symbol
nonyl phenyl eicosaethylene glycol ether	NPEGE
nonyl phenyl triaconta ethylene glycol ether	NPTGE
nonyl phenyl tetraconta ethylene glycol ether	NPTTGE
nonyl phenyl pentacona ethylene glycol ether	NP50E
nonyl phenyl decaconta ethylene glycol ether	NP100E
polyoxyethylene-23-sorbitan monolaurate	PSML

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